Thermal Pasteurization Effects on Color of Red Grapefruit Juices

H.S. Lee and G.A. Coates

ABSTRACT

Changes in color due to thermal pasteurization of two cultivars of red grapefruit juice were studied. Juices were pasteurized at 91°C using a plate heat exchanger. Apparently, thermal pasteurization changed all three color parameters (CIE L*, a*, b*) in the juice, causing a slight color shift towards lighter and brighter. Thermal pasteurization especially affected CIE b* value and chroma in juice. The reflectance spectrum in the visible region (400 nm to 700 nm) clearly showed changes in spectral distribution of light reflected from juice after pasteurization. There were no changes ($P>0.05$) in major carotenoid pigments (β-carotene and lycopene) in the juices after pasteurization.

Key Words: red grapefruit juice, pasteurization, color, pigments, carotenoid

INTRODUCTION

THE RED CULTIVARS OF GRAPEFRUIT ARE AN IMPORTANT AND WIDELY planted citrus crop. Red grapefruit has a pleasing visual appearance and mild taste, and there has been considerable demand for fresh as well as processed colored grapefruit products, especially for beverages and cocktail preparations (Labell, 1993). The flesh of the red grapefruit contains high amounts of carotenoid pigments. Lycopene and ß-carotene have long been known to be principal pigments in red grapefruit with lesser amounts of phytofluene and zeta-carotene (Curl and Bailey, 1957). The pink to red color in red grapefruit is primarily associated with lycopene (Ting and Deszyck, 1958). Most published studies on color of red grapefruit considered pigmentation changes with maturity (Lime et al.,1954), cultivar (Cruse et al., 1979), quantitative differences (Rouseff et al.,1992), and seasonal changes (Ting et al., 1980), as well as factors affecting pigmentation and color in processed products (Lee, 1997).

Carotenoids are highly unsaturated compounds and therefore susceptible to oxidation, isomerization, and other chemical changes during processing and storage (Boskovic, 1979). The stability of carotenoids in foods varies greatly; some high-carotenoid vegetables show a distinct color shift when heated in water, while others do not (Purcell et al., 1969). Red pigmented grapefruits often yield a juice product with a color that is neither distinctive nor consumer acceptable (Huggart et al., 1979), since colored grapefruit juice is sensitive to heat (Ting et al., 1980). The pink to red color due to lycopene is somewhat unstable during processing and storage, and the juice can develop a muddy, brown unacceptable color (Shaw and Nagy, 1993). Detailed knowledge of pigment behavior during processing and its effects on visual color can help in improving color and its stability in juice products.

This study was a part of a broader grapefruit juice quality improvement program and was initiated to evaluate any visual color changes associated with thermal processing of juice products from red grapefruit cultivars. Our objective was to compare the changes and stability of juice color during thermal pasteurization between the lightly colored Ruby Red, and the more intensely colored Star Ruby.

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MATERIALS & METHODS

RUBY RED AND STAR RUBY GRAPEFRUITS (*CITRUS PARADISI MACFAD.*) from Indian River and central Florida growing regions were used. The juices were prepared using commercial FMC juice extractors using standard settings and finished in an FMC juice finisher in the pilot plant at the Citrus Research & Education Center (Univ. of Florida, Lake Alfred, FL). The finished juice was pumped to a holding tank prior to pasteurization. Thermal pasteurization was performed by pumping the finished juice through an APV (Tonawanda, NY) plate heat exchanger. The juice was heated to 91°C at a flow rate of 3.8 L per minute (ca 10 sec) and followed by rapid chilling to 25°C . Juice samples were packed in 950 mL high density polyethylene (HDPE) bottles. The same treatment was applied to the juices throughout the processing season from Nov. 1996 to May 1997. Both fresh (collected before pasteurization) and pasteurized juices were analyzed for color and pigment contents on the same day as processed.

Color analysis

Color was measured on duplicate samples in test tubes (25 mm × 20 cm, o.d.). The CIE L*, a*, b* values were measured with a Macbeth COLOR-EYE© 3100 spectrophotometer (Kollmorgen Instruments Corp., Newburgh, NY) with Optiview software package in the reflectance mode, with illuminant C and 2° observer angle. From CIE a* and b* values the chroma $\{(a^{*2}+b^{*2})^{1/2}\}$, and hue angle ($\tan^{-1} b^*/a^*$) were calculated. Total color differences, ΔE^* before and after pasteurization, were calculated using the (L*, a*, b*) color coordinates as defined by the equation: $(\Delta L^{*2}+\Delta a^{*2}+\Delta b^{*2})^{1/2}$. Differences in CIE L*, a*, b* values between replicates were <1%.

Pigment analysis by HPLC

Pigment analysis was conducted using a previously described HPLC method (Sadler et al., 1990; Sander et al., 1994) with modification. Grapefruit juice (2 mL) was mixed with 5 mL of hexane-ethanol-acetone (50:25:25), agitated, and centrifuged for 5 min at 6,500 rpm in a refrigerated centrifuge (model MP4R, International Equipment Company, Needham Heights, MA) at 5°C. The solution separated into distinct polar and nonpolar layers. The upper hexane layer was used for pigment analysis. Reproducibility of analysis was <1% CV for the six runs of extracts prepared from the same juice.

HPLC system consisted of a Waters 600E gradient pump and a 717 plus autosampler equipped with chiller (Waters Associates, Milford, MA). Analyses were carried out using a YMC (Wilmington, NC) C_{30} column (4.6 mm × 15 cm, 3 μm), oven temperature at 25°C, using binary gradient elution. The eluents were methanol (A) and methyl-t-butyl ether (B). Both eluents contained 0.05% triethylamine and 0.01% BHT. The gradient program (linear step) was 75% A/25% B initial, to 65% A/ 35% B in 10 min, to 45% A/55% B in 10 min, isocratically run for 5 min, and then returned to the initial condition. The flow rate was 1.5 mL/min, and injection volume was 10 μL. For detection, a Spectra- Physics (Riviera Beach, FL) UV-Visible detector and a Waters 996 photodiode-array detector were used. β-carotene and lycopene were identified by retention times and by comparison of visible spectra with those of pure compounds. Standards of carotenoids were obtained from Sigma Chemical Co. (St. Louis, MO). All data acquisition and processing were done using *Millennium Chromatography software (version 2.1*) from Waters. All data were duplicate analyses and mean values were reported.

Statistical analysis

Statistical analysis was conducted using the SigmaStat PC software from SPSS, Inc (Chicago, IL). Trends were considered significant when means of compared sets differed at P<0.05 (Student's t-test).

RESULTS & DISCUSSION

Effects on color coordinates

A total of 15 juice samples, including two red cultivars of grapefruit (Ruby Red and Star Ruby) juice, were processed through the 1996–97 processing season to evaluate thermal pasteurization effects on color changes. Slight visual differences were perceived after thermal pasteurization, and color values were compared in both fresh and pasteurized juices (Table 1). Most samples showed slight increases in L* value after pasteurization, which indicated a lightening of juice surface color. A small increase in L* value for pasteurized juices could probably be attributed to partial precipitation of unstable, suspended particles in the juice (Genovese et al., 1997). They reported a similar observation of small increases in L* values after thermal processing of cloudy apple juice. Precipitation of insoluble particles from cloudy apple juice suspension was reported to contribute to increases in lightness after pasteurization.

Color differences in fresh and pasteurized juices were also plotted on a chroma diagram (a* versus b*) (Fig. 1). Clearly illustrated are the changes of CIE a* and b* parameters after pasteurization, especially large changes were found in the b* value (P<0.05). After thermal treatment, b* values gradually changed toward more positive values in all samples. Changes in a* followed a similar trend, shifting toward positive values. The changes in a* value, however, were small (P>0.05) as compared to b*, and may not contribute significantly to perception of color change. Results suggest that color differences were more related to changes in b* value, with yellowing of samples as the most noticeable visual change produced by pasteurization. A color shift toward positive b* and positive a* directions (Fig. 1) indicated more yellow and more red in the pasteurized juices.

Also, CIE L*, a*, b* values were used to calculate total color differences (ΔE^*) after pasteurization, which indicated the magnitude of overall color difference between fresh and pasteurized juices (Table 1). The ΔE^* values ranged from 2.5 to 4.8 for Ruby Red juices. However, note that the magnitude of color difference appeared to be smaller in highly pigmented Star Ruby juices compared to Ruby Red. A similar result has been observed from previous work with red grapefruit juices during a storage test in our lab; color differences due to storage-aging were smaller with higher pigment content in the juice (Lee, 1997). This indicates using highly pigmented fruits would provide a better masking effect on color changes and probably result in more acceptable juice color after processing and storage.

In the interpretation of color differences, Francis and Clydesdale (1975) indicated that a ΔE^* of 2 would be noticeably different and a ΔE^* of 3 would be unacceptable for many products. In all tested juices, the total color differences between before and after pasteurization were higher than ΔE^* of 2 (Table 1), confirming the noticeable visual difference. Interpreting the magnitude of color differences obviously differs with each type of application and commodity. However, of 15 samples, four juices had ΔE^* greater than 3, which is probably an unacceptable visual change after pasteurization but thermal pasteurization is necessary for most commercial juice products.

Effects on hue and chroma

The range for the hue angle of eight fresh Ruby Red juices was from 83.55 to 103.12. The hue angle for seven fresh Star Ruby juices was 31.46 to 58.94 (Fig.1). Star Ruby fruits were visibly more intensely colored (more red) than Ruby Red. After pasteurization, the hue angle changed in both cultivars but the magnitude of hue shift was not large (P>0.05). Ting et al. (1980) had reported a visible color change by heating Star Ruby grapefruit juice, which caused a substantial decrease of the Hunter a/b, but retained considerable color after pasteurization and concentration.

Chroma, which represents color intensity, increased after pasteurization (P<0.05). Both hue angle and chroma increased after pasteurization of Star Ruby juice. While hue angle decreased in most Ruby Red juices (6 of 8), chroma increased. Since hue angle is a function of a* and b* values, it may be due to the negative a* values in those six fresh juices. There is relatively little published information relating consumer acceptance of color with red grapefruit juices.

We compared differences in lightness (ΔL^*) and chroma (ΔC^*) before and after pasteurization (Fig. 2). The confidence ellipse (p=0.95) was centered on the sample means of the *x* (ΔC^*) and *y* (ΔL^*) variables. This data indicate the direction of color difference in juices after pasteurization but do not describe the degrees of color differences. After pasteurization, juice color shifted toward the direction between positive ΔC^* and positive ΔL^*, indicating the color of pasteurized juice became slightly lighter and brighter than fresh juice. A similar observation of increases in brightness (increase in chroma) due to application of heat during the manufacture of tomato juice has been long known.

Effects on reflectance curve

Reflectance curves of Ruby Red grapefruit juices (Fig. 3) showed differences in the magnitude of light reflected but patterns were similar in Ruby Red and Star Ruby juice. Upon pasteurization, the reflectance spectrum changed (P<0.05). After pasteurization, the reflectance in the 420 nm to 520 nm range decreased, and above 520 nm increased. A decrease in reflection (increase in absorption) could be attributable to a general hyperchromic effect throughout the visible region of the spectra below 520 nm, particularly throughout the blue-green portions (420 nm to 520 nm). There were more changes throughout the visible region above 520 nm, indicating relatively more yellow-red light would be reflected to the eye and samples would appear more yellow-red.

Effects on pigment contents

The major carotenoid pigments, ß-carotene and lycopene, responsible for visual color of the fresh and pasteurized juices, were compared by HPLC (Table 1). Total pigment content, the sum of the two major pigments β-carotene and lycopene, ranged from 1.6 to 3.3 ppm for fresh Ruby Red juices. In the fresh Star Ruby juices, total pigment was three to 14 times greater, ranging from 10.8 to 22.8 ppm. As previously reported (Cruse et al.,1979), variation in amounts of carotenoids, especially red lycopene in Star Ruby juices, was observed as

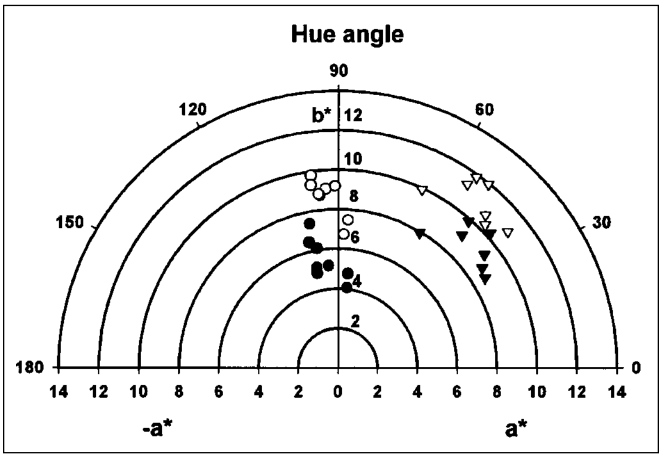

Fig. 1—Changes in CIE a* and b* values after thermal pasteurization. Ruby Red (fresh, ●; pasteurized, ○) and Star Ruby (fresh, ▼; pasteurized, ▽) grapefruit juices.

Table 1—Color and pigment contents in red cultivars of grapefruit juices

Juices	Date	L*	a*	b*	Chroma (°C)	Hue (H*)	ΔE*	β-carotene (ppm)	Lycopene (ppm)	Total (ppm)
Ruby Red										
RR1-Fresh	11/14/96	39.01	0.46	4.04	4.07	83.55		1.0	2.4	3.4
RR1-Past.	11/14/96	39.50	0.28	6.73	6.74	87.59	2.7	1.0	2.2	3.2
RR2-Fresh	12/13/96	37.93	0.49	4.73	4.76	84.04		1.0	2.2	3.2
RR2-Past.	12/13/96	38.95	0.51	7.45	7.47	86.08	2.9	1.1	2.5	3.6
RR3-Fresh	12/13/96	39.60	-0.48	5.14	5.16	95.34		1.1	1.4	2.5
RR3-Past.	12/13/96	41.03	-0.16	9.17	9.17	90.99	4.3	1.1	1.2	2.3
RR4-Fresh	1/13/97	38.12	-1.05	4.75	4.87	102.40		1.2	1.2	2.4
RR4-Past.	1/13/97	40.18	-0.64	9.02	9.04	94.04	4.8	1.3	1.3	2.6
RR5-Fresh	2/19/97	39.70	-1.06	5.04	5.15	101.91		1.1	0.9	2.0
RR5-Past.	2/19/97	39.95	-0.92	8.68	8.73	96.02	3.7	1.3	0.9	2.2
RR6-Fresh	3/18/97	39.95	-1.08	6.01	6.10	100.18		1.0	0.9	1.9
RR6-Past.	3/18/97	40.82	-1.00	8.76	8.81	96.49	2.9	1.1	0.9	2.0
RR7-Fresh	4/25/97	41.58	-1.44	7.26	7.40	101.24		1.3	0.8	2.1
RR7-Past.	4/25/97	42.24	-1.39	9.69	9.79	98.18	2.5	1.1	0.8	1.9
RR8-Fresh	5/20/97	41.11	-1.47	6.30	6.47	103.12		1.0	0.7	1.7
RR8-Past.	5/20/97	41.81	-1.39	9.21	9.31	98.57	3.0	1.0	0.7	1.7
Star Ruby										
SR1-Fresh	11/13/96	32.66	7.40	4.53	8.68	31.46		1.6	15.8	17.4
SR1-Past.	11/13/96	34.34	8.53	6.86	10.95	38.80	3.1	1.6	15.9	17.5
SR2-Fresh	12/13/96	33.75	7.25	5.02	8.82	34.70		2.9	18.4	21.3
SR2-Past.	12/13/96	34.48	7.40	7.20	10.33	44.22	2.3	2.7	16.5	19.2
SR3-Fresh	1/13/97	33.80	7.35	5.68	9.29	37.71		3.0	19.8	22.8
SR3-Past.	1/13/97	34.46	7.42	7.71	10.70	46.09	2.1	3.2	20.5	23.7
SR4-Fresh	2/19/97	34.41	7.68	6.79	10.25	41.50		4.5	13.5	18.0
SR4-Past.	2/19/97	34.83	7.56	9.26	11.96	50.77	2.5	4.1	12.6	16.7
SR5-Fresh	3/18/97	34.90	6.23	6.68	9.13	47.00		4.8	12.8	17.6
SR5-Past.	3/18/97	35.27	6.97	9.61	11.87	54.05	3.0	5.3	14.8	20.1
SR6-Fresh	4/25/97	34.66	6.57	7.39	9.88	48.37		4.1	16.5	20.6
SR6-Past.	4/25/97	35.78	6.51	9.27	11.33	54.92	2.2	3.9	14.8	18.7
SR7-Fresh	5/20/97	36.06	4.10	6.80	7.94	58.94		2.0	8.7	10.7
SR7-Past.	5/20/97	37.12	4.21	9.00	9.93	64.90	2.4	2.1	8.7	10.9

the season progressed (Table 1). Lycopene is the major colored pigment in Star Ruby with lesser β-carotene. Most of the Ruby Red juices (4 of 8) contained slightly higher amounts of β-carotene than lycopene.

There were no changes in β-carotene or lycopene contents after thermal pasteurization that were significant ($P>0.05$). Carotenoids are generally stable to heat treatment involved in common unit operations of food processing such as blanching, cooking and canning (Borenstein and Bunnell, 1967), but are rapidly lost on dehydration (Simpson, 1985). Specifically, the thermal stability of lycopene within plant tissue has been stressed in studies on processed fruits and vegetables (Nguyen and Schwartz, 1998; Schwartz, 1998). Under this pasteurization condition (91±1°C, ca 10 sec), no clear differences in thermal stability of β-carotene and lycopene were observed. The normal pasteurization conditions for citrus juices can vary depending on containers and the storage environment; ranging between 74°C and 99°C for 2 sec to 16 sec (Fellers, 1991). Thermal pasteurization did not cause notable pigment loss under the current processing conditions compared to previous work with orange juice (Lessin et al.,1997). In that study with various fruits and vegetables, there were quantitative losses of carotenoids in pasteurized orange juice after 2 min heating at 80°C due to trans to cis isomerization. Geometric isomers of β-carotene and lycopene in red grapefruit juice were tentatively characterized based on spectra by HPLC-PDA, and determined to be traces by HPLC. Furthermore, thermal trans-cis isomerization of lycopene was reported to be strictly limited to drying stages (Boskovic, 1979) but their quantification was not attempted for our study.

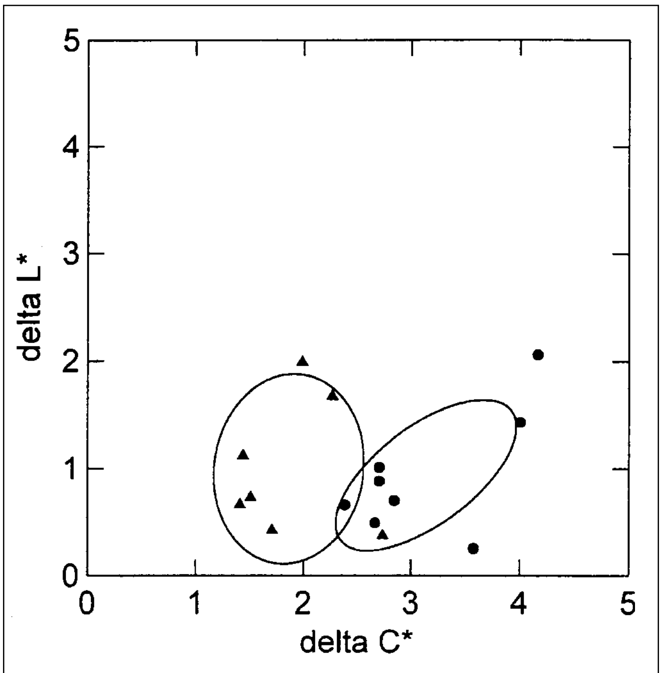

Fig. 2—Differences in lightness and chroma (ΔC* versus ΔL*) in juices after thermal pasteurization. Ruby red (●) and Star Ruby (▼) grapefruit juices.

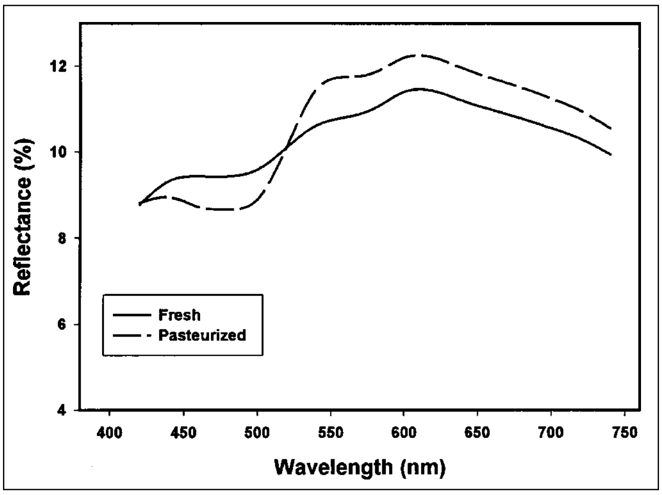

Fig. 3—Spectral reflectance graphs of fresh and pasteurized Ruby Red grapefruit juices.

The color changes cannot be explained by a change in carotenoid content nor isomerization to cis-carotenoid (Purcell et al., 1969). A change in physical state of the carotenoid probably is responsible for heat-caused color changes. The color shift during thermal processing was attributed to degradation of chromoplasts and solution of carotenes in other cellular lipids. Furthermore, Genovese et al. (1997) speculated that since juice color was reflected by suspended pulp particles (juice sacs), changes in suspended pulp particles after thermal pasteurization probably would also affect color changes in juices.

CONCLUSIONS

THERE WAS PERCEPTIBLE COLOR CHANGE DURING PASTEURIZATION of juice from red grapefruit, which led to juice color becoming lighter and more saturated. Large changes in the CIE b* value and chroma (saturation) suggested that these values would be better indicators representing color changes than other color parameters for pasteurized red grapefruit juice. Overall increases in reflected light and hyperchromic effect, mainly in the blue-green portion of the spectra, might influence perception of color to a great extent in pasteurized juice. Thermal effects on lycopene and β-carotene pigment contents were not clearly detected. However, any color changes after pasteurization were less perceptible visually with highly pigmented juices.

REFERENCES

Boskovic, M. A. 1979. Fate of lycopene in dehydrated tomato products: Carotenoid isomerization in food system. J. Food Sci. 44: 84-86.

Borenstein, B. and Bunnell, R.H. 1966. Carotenoids: properties, occurrence, and utilization in foods. In *Advances in Food Research*, C.O. Chichester, E.M. Mrak, and G.F. Stewart (Ed.). Vol 15: p. 195-176. Academic Press, New York.

Cruse, R.R., Lime, B.J., and Hensz, R.A. 1979. Pigmentation and color comparison of Ruby Red and Star Ruby grapefruit juice. J. Agric. Food Chem. 27: 641-642.

Curl, A.L. and Bailey, G.F. 1957. The carotenoids of Ruby Red grapefruit. Food Res. 22: 63-68.

Fellers, P.J. 1991. Citrus Industry. In *Encyclopedia of Food Science & Technology*, Y.H. Hui (Ed.), Vol. 1, p. 420-438. John Wiley & Sons, Inc., New York.

Francis, F.J. and Clydesdale, F.M. 1975. *Food colorimetry: Theory and Applications*. The AVI Publishing Co., Inc., Westport, CT.

Genovese, D.B., Elustondo, M.P., and Lozano, J.E. 1997. Color and cloud stabilization in cloudy apple juice by steam heating during crushing. J. Food Sci. 62: 1171-1175.

Huggart, R.L., Fellers, P.J., Jager, G.D., and Brady, J. 1979. The influence of color on consumer preferences for Florida frozen concentrated grapefruit juices. Proc. Fla. State Hort. Soc. 92: 148-151.

Labell, F. 1993. Pink grapefruit beverages: Mainstream refreshment. Food Process. 54(5): 66.

Lee, H.S. 1997. Issue of color in pigmented grapefruit juice. Fruit Process. 7(4): 132-135.

Lessin, W.J., Catigani, G.L., and Schwartz, S.J. 1997. Quantification of cis-trans isomers of provitamin A carotenoids in fresh and processed fruits and vegetables. J. Agric. Food Chem. 45: 3728-3732.

Lime, B.J., Stephens, T.S., and Griffiths, F.P. 1954. Processing characteristics of colored Texas grapefruit. 1. Color and maturity studies of Ruby Red grapefruit. Food Technol. 8(12): 566-569.

Nguyen, M.L. and Schwartz, S.J. 1998. Effects of industrial thermal treatments on carotenoid geometrical isomers in fresh and processed fruits and vegetables. Presented at Ann. Mtg., Inst. of Food Technologists, Atlanta, GA, June 20-24.

Purcell, A.E., Walter, W.M., Jr., and Thompkins, W.T. 1969. Relationship of vegetable color to physical state of the carotenes. J. Agric. Food Chem. 17: 41-42.

Rouseff, R.L., Sadler, G.D., Putnam, T.J., and Davis, J.E. 1992. Determination of ß-carotene and other hydrocarbon carotenoids in red grapefruit cultivars. J. Agric. Food Chem. 40: 47-51.

Sadler, G.D., Davis, J., and Dezman, D. 1990. Rapid extraction of lycopene and ß-carotene from reconstituted tomato paste and pink grapefruit homogenates. J. Food Sci. 55: 1460-1461.

Sander, L.C., Sharpless, K.E., Craft, N.E., and Wise, S.A. 1994. Development of engineered stationary phases for the separation of carotenoid isomers. Anal. Chem. 66: 1667-1674.

Schwartz, S.J. 1998. Lycopene stability during food processing. Presented at Ann. Mtg., Inst. of Food Technologists, Atlanta, GA, June 20-24.

Shaw, P.E. and Nagy, S. 1993. Grapefruit juice. Ch. 6 in *Fruit juice processing technology*, S. Nagy, C.S. Chen, and P.E. Shaw (Ed.), p. 166-214. AgScience, Inc., Auburndale, FL.

Simpson, K.L. 1985. Chemical changes in natural food pigments. Ch.18 in *Chemical changes in food during processing*, T. Richardson, and J. W. Finley (Ed.), p. 409-441. AVI Pub. Co., Inc., Westport, CT.

Ting, S.V. and Deszyck, E.J. 1958. The internal color and carotenoid pigments of Florida red and pink grapefruit. Proc. Am. Soc. Hort. Sci. 71: 271-277.

Ting, S.V. and Hendrickson, R. 1968. Enhancing color of orange juice with natural pigments from orange peel. Proc. Fla. State Hort. Soc. 81: 264-268.

Ting, S.V., Huggart, R.L., and Ismail, M.A. 1980. Color and processing characteristics of Star Ruby grapefruit. Proc. Fla. State Hort. Soc. 93: 293-295.

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